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Scattering of X-Rays by Metals at Very Small Angles

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The scattering of x-rays at very small angles ($\varphi <$ 1° or 2°) to the incident beam is due to submicroscopic regions in the scattering substance having different electron densities. The fluctuations of the electron density in a solid and in particles which are distributed at distances greater than their own dimensions cause a "gas-type" scattering, characterized by a monotonically decreasing intensity as the angle φ increases; on the other hand, tightly-packed particles cause a "liquid-type" scattering, whose pattern is a diffused ring with a diameter determined by the average distance between the scattering centers.

The theory of the scattering of x-rays at very small angles,[1,2] based on the theory of scattering by gases and liquids,[2] has not yet been fully developed. However, a large number of investigations in this field, concerned with determining the size and shape of particles of various substances and their submicroscopic defects,[3] have given quite satisfactory results. This fact served as a basis for the study of scattering by pure metals, described in this paper.

(1) In order to obtain a picture of scattering at very small angles ($\varphi <$ 10'), we assembled an apparatus with two slits 0.014 mm wide and 60 mm apart. We found that the intensity distribution across the incident beam passing through the slits was of the type e^{-ax^2} and that its angular width φ (between the points of half-maximum intensity) was about 25". Such a distribution of intensity, resulting probably from the profile of the slits and the passage of the rays through their edges, offers many opportunities for studying the scattering at very small angles.

In our work we used the radiation from an x-ray tube having a molybdenum anode and operated at the stabilized voltage of 37 kv; this voltage provides an advantageous ratio between the intensity of the monochromatic radiation and the accompanying white radiation.

Short-wave radiation, which scatters within a smaller range of angles φ than long-wave radiation, was used because of the high absorption of the latter.

In order to obtain the scattering pattern at very small angles, the x-ray plate was placed 170 cm from the sample. At this distance, the layer of air and the metallic sample itself weaken sufficiently the long-wave part of the white radiation, which introduces the largest error in determining the lower limit of the size of the particles (20 to 50 percent),[4] usually found from the approximate formula

$$I_\varphi = N n^2 e^{-\frac{4}{5}\pi^2\left(\frac{R}{\lambda}\right)^2\varphi^2}$$

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Here I_ϕ is the intensity of the radiation scattered at the angle φ, N is the number of the scattering particles or of defects of size R, and n is the number of electrons in one particle.

(2) We studied samples of pure electrolytic copper rolled into sheets 0.11 mm thick, some not annealed, others annealed *in vacuo* at 800 degrees for 4 hours; samples of very pure cast aluminum (99.99 percent) 1.5 mm thick; similar samples of cold-worked aluminum; and samples of aluminum of commercial purity, prepared by sintering.

Fig. 1. X-ray photographs (photographic enlargement 1.5). *a*, direct beam without sample; *b*, sample of rolled copper, axis of rolling parallel to slit; *c*, axis of rolling perpendicular to slit; *d*, annealed sample of rolled copper, axis of rolling parallel to slit; *e*, axis perpendicular to slit.

Fig. 1 shows x-ray photographs of the direct beam of rays taken without a sample and with copper samples exposed with the axis of rolling parallel and perpendicular to the slit. They distinctly show the diffusion of the incident beam, consisting in the widening of the beam and the monotonic decrease of intensity, which extends as a weak background into a relatively large angular range.

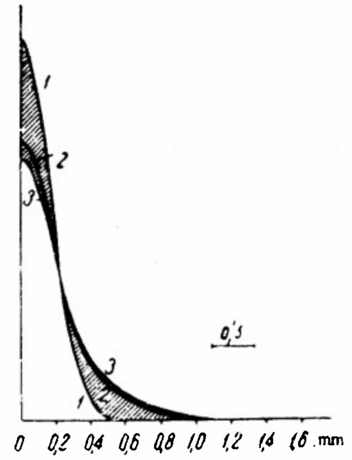

Fig. 2. Microphotometer curves. *1*, direct beam without sample; *2*, recrystallized copper; *3*, cold-worked copper.

Figs. 2 and 3 show the microphotometer curves giving the angular dependence of the intensity of the direct beam *1* and of the beams *2*, *3*, and *4* that have passed through the samples. The areas under these curves are equal because the ordinates have been multiplied by the proper coefficients. Thus, the effect of the absorption μ is practically eliminated, and the areas bordered by the curves *1* and *2* or *3* and *4* (cross-hatched in Fig. 2) are determined by the additional absorption μ' due to the scattering at small angles, which, as has been shown by Warren,[6] is proportional to the radius of the defects or of the particles and to the density ρ within these regions; in fact, $\mu' = 0.108\,\lambda^2 R\rho$.

(3) The x-ray photographs and the distribution curves of the intensity of scattering at very small angles show that pure metals, whether deformed, cast, recrystallized, or obtained by sintering, give a "gas scattering" pattern. Our interpretation of this pattern is that the scattering results from the difference in electron densities in the metal itself and in the defects (micropores and cracks), which are voids with zero electron density. This interpretation is based particularly on the fact that the scattering by porous aluminum (and other metals) obtained by sintering becomes particularly conspicuous, judging by the decrease of the area bordered in Fig. 3 by the curves *1* and *4* before their intersection. The

fact that rolled copper scatters more and at larger angles φ when the rolling axis of the samples is parallel to the slit than when it is perpendicular shows that the defects are elongated and are oriented in the direction of the axis of rolling. This phenomenon agrees with the concepts concerning the nature of the strength of metals.[6]

The data on the scattering by rolled and recrystallized copper show that the defects (the lower limit of whose size is estimated as 300 A in the case of a cold-worked metal) grow to 400 A upon recrystallization, probably because of the general lowering of the saturation of the copper by smaller defects, and because of the decrease in the randomness of the orientation of their axes.

In cast aluminum, the lower limit of the size of the defects is about 300 A. Under plastic deformation, this limit is reduced in aluminum to about 250 A with the general decrease of saturation of the metal by the larger defects, probably due to their healing.

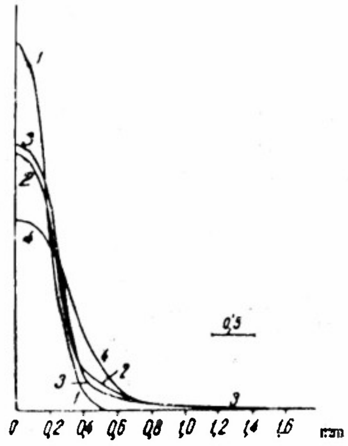

Fig. 3. Microphotometer curves. 1, direct beam without sample; 2, cast aluminum; 3, cold-worked aluminum; 4, aluminum obtained by sintering.

In the aluminum obtained by sintering, the lower limit of the size of the defects coincides with the limit for cast aluminum. The former differs from the latter by a greater saturation with defects of a considerable size, probably more than 1000 A.

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